

Appl. No. 10/642,805  
Amdt. dated September 6, 2005  
Reply to Office Action of May 3, 2005

Amendments to the Claims:

This listing of claims will replace all prior versions, and listings, of claims in the application:

Listing of Claims:

Claim 1 (currently amended). A method of making a catalyst for the production of hydrogen, said catalyst comprising from about 60 wt% to about 95 wt% Fe<sub>2</sub>O<sub>3</sub>, and from about 0.1 wt% to about 20 wt% chromium in the form of the oxide Cr<sub>2</sub>O<sub>3</sub>, and from about 0 wt% to about 10 wt% CuO, and said method comprising:

- a) preparing an aqueous organic acid solution, wherein said organic acid is a carboxylic acid having at least one carboxylic acid group with a pK<sub>a</sub> at ambient temperature of from about 0.5 to about 6;
- b) adding iron metal to said acid solution;
- c) forcing an oxidative oxidizing agent through said acid solution until said iron metal is consumed and an iron slurry is formed;
- d) milling said iron slurry to a particle size with a D<sub>50</sub> of less than about 2 microns;
- e) adding at least one promoter to said milled iron slurry to form a product slurry, said promoter being added at a concentration such that said product slurry has a solids content of from about 10 % to about 40 % inclusive of said promoter;
- f) drying said slurry to form particles;
- g) optionally, mixing in chromic acid flakes to provide up to about 20 wt% Cr<sub>2</sub>O<sub>3</sub> to the finished catalyst; and
- g) calcining said particles to form said catalyst.

Claim 2 (original). The method of Claim 1 wherein said organic acid is selected from the group consisting of formic acid, acetic acid, glycolic acid, oxalic acid, pyruvic acid, malonic acid and propionic acid, and a combination thereof.

Claim 3 (original). The method of Claim 1 wherein said iron metal is a powder, granule, sphere, chip or other form having an average diameter of from about 1μ to about 500μ.

Claim 4 (original). The method of Claim 1 wherein said promoter is selected from the group consisting of cerium, chromium, iridium, lanthanum, manganese, molybdenum, palladium, platinum, rhenium, rhodium, ruthenium, strontium, tungsten, vanadium, zinc, potassium oxide, rubidium oxide, cesium oxide, magnesium oxide, titanium oxide, zirconium oxide, aluminum oxide, silica, scandium,

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yttrium, praseodymium, neodymium, promethium, samarium, europium, gadolinium, terbium, dysprosium, holmium, erbium, thulium, ytterbium, lutetium, other rare earth metals and combinations thereof.

Claim 5 (original). The method of Claim 1 wherein said slurry is spray dried with a wheel atomizer.

Claim 6 (original). The method of Claim 1 wherein said catalyst comprises from about 80 wt % to about 95 wt % iron oxide.

Claim 7 (original). The method of Claim 1 wherein said catalyst has an essentially spherical particle shape and relatively small particle size distribution range.

Claim 8 (original). The method of Claim 1 wherein said oxidizing agent is air, compressed air, oxygen, hydrogen peroxide, an organic peroxide, ozone and a combination thereof.

Claim 9 (currently amended). The method of Claim 1 wherein said catalyst further comprises from about 0 wt% to about 10 wt% of a component selected from the group consisting of CuO, ZrO<sub>2</sub>, TiO<sub>2</sub>, Co<sub>3</sub>O<sub>4</sub>, Al<sub>2</sub>O<sub>3</sub>, SiO<sub>2</sub> and CeO<sub>2</sub>, and said component is added to said milled iron slurry with said promoter.

Claim 10 (currently amended). A method of making a catalyst for the production of hydrogen, said catalyst comprising from about 60 wt% to about 95 wt% Fe<sub>2</sub>O<sub>3</sub>, from about 0 0.1 wt% to about 20 wt% chromium in the form of the oxide Cr<sub>2</sub>O<sub>3</sub>, and from about 0 wt% up to about 10 wt% CuO, and said method comprising:

- a) preparing an aqueous organic acid solution, wherein said organic acid is a carboxylic acid having at least one carboxylic acid group with a pK<sub>a</sub> at ambient temperature of from about 0.5 to about 6;
- b) adding an iron source to said acid solution, wherein said iron source is an iron metal powder, granule, sphere, chip or other form having an average diameter of from about 1μ to about 500μ;
- c) forcing an oxidative oxidizing agent through said acid solution until said iron source is consumed and an iron slurry is formed;
- d) milling said iron slurry to a particle size with a D<sub>50</sub> of less than about 2 microns;
- e) adding said CuO and, optionally, at least one promoter, to said milled iron slurry to form a product slurry, said CuO and optional promoter being added at a concentration such

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- that said product slurry has a solids content of from about 10 % to about 40 % inclusive of said promoter;
- f) drying said slurry to form particles;
  - g) mixing in chromic acid flakes to provide up to about 20 wt% Cr<sub>2</sub>O<sub>3</sub> to the finished catalyst; and
  - h) calcining said particles to form said catalyst.

Claim 11 (original). The method of Claim 10 wherein said milled slurry is spray dried with a wheel atomizer.

Claim 12 (original). The method of Claim 10 wherein said iron slurry has a solids content of from about 10 % to about 40 %.

Claim 13 (original). The method of Claim 10 wherein said organic acid solution is prepared from water and an acid selected from the group consisting of formic acid, acetic acid, glycolic acid, oxalic acid, pyruvic acid, malonic acid and propionic acid, and a combination thereof.

Claim 14 (original). The method of Claim 10 wherein said oxidizing agent is air, compressed air, oxygen, hydrogen peroxide, an organic peroxide, ozone and a combination thereof.

Claim 15 (original). The method of Claim 10 wherein said promoter is selected from the group consisting of cerium, chromium, iridium, lanthanum, manganese, molybdenum, palladium, platinum, rhenium, rhodium, ruthenium, strontium, tungsten, vanadium, zinc, potassium oxide, rubidium oxide, cesium oxide, magnesium oxide, titanium oxide, zirconium oxide, aluminum oxide, silica, scandium, yttrium, praseodymium, neodymium, promethium, samarium, europium, gadolinium, terbium, dysprosium, holmium, erbium, thulium, ytterbium, lutetium, other rare earth metals and combinations thereof.

Claim 16 (currently amended). The method of Claim 10 wherein said catalyst further comprises from about 0 wt% to about 10 wt% of a component selected from the group consisting of ZrO<sub>2</sub>, TiO<sub>2</sub>, Co<sub>3</sub>O<sub>4</sub>, Al<sub>2</sub>O<sub>3</sub>, SiO<sub>2</sub> and CeO<sub>2</sub>, and said component is added to said milled iron slurry with said promoter.

Claim 17 (currently amended). A catalyst for the production of hydrogen, said catalyst being prepared by reacting an essentially contaminant-free iron source with an organic acid and air to form an iron oxide

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slurry, adding one or more promoters to said iron oxide slurry to form a promoter slurry, spray drying said promoter slurry, adding a chromium source to form a product mix, and calcining said product mix, and wherein said catalyst comprises from about 60 wt% to about 95 wt%  $\text{Fe}_2\text{O}_3$ , and from about 0.1 wt% to about 20 wt% chromium in the form of the oxide  $\text{Cr}_2\text{O}_3$ , ~~and from about 0 wt% to about 10 wt%  $\text{CuO}$ .~~

Claim 18 (currently amended). The catalyst of Claim 17 wherein said catalyst further comprises from about 0 wt% to about 10 wt% of a component selected from the group consisting of  $\text{CuO}$ ,  $\text{ZrO}_2$ ,  $\text{TiO}_2$ ,  $\text{Co}_3\text{O}_4$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{SiO}_2$  and  $\text{CeO}_2$ , and said component is added to said milled iron slurry with said promoter.

Claim 19 (original). The catalyst of Claim 17 wherein said essentially contaminant-free iron slurry is prepared by reacting an iron source selected from an iron metal powder, a granule, a sphere, a chip or a particle having an average diameter of from about  $1\mu$  to about  $500\mu$ , with an aqueous organic acid solution and an oxidizing agent.

Claim 20 (original). The catalyst of Claim 17 wherein said organic acid solution is prepared from water and a carboxylic acid having at least one carboxylic acid group with a pK<sub>a</sub> at ambient temperature of from about 0.5 to about 6, and said oxidizing agent is air, compressed air, oxygen, hydrogen peroxide, an organic peroxide, ozone and a combination thereof.

Claim 21 (original). The catalyst of Claim 17 wherein said promoter is selected from the group consisting of cerium, chromium, iridium, lanthanum, manganese, molybdenum, palladium, platinum, rhenium, rhodium, ruthenium, strontium, tungsten, vanadium, zinc, potassium oxide, rubidium oxide, cesium oxide, magnesium oxide, titanium oxide, zirconium oxide, aluminum oxide, silica, scandium, yttrium, praseodymium, neodymium, promethium, samarium, europium, gadolinium, terbium, dysprosium, holmium, erbium, thulium, ytterbium, lutetium, other rare earth metals and combinations thereof.